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ARMOUR RESEARCH FOUNDATION
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ILLINOIS INSTITUTE OF TECHNOLOGY
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Project No. ARF-B224
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CO-REDUCTION AND CONSOLIDATION
OF Mo-0.5Ti-0.1C ALLOY

Report No. ARF-B224-5
(Final Report)
April 27, 1961 - October 31, 1962

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Department of the Navy
Bureau of Naval Weapons
Materials Division
Washington 25, D. C.

February 4, 1963

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ABSTRACT

Co-reduction of preblended halides by a modified Kroll Process has been investigated as a means of producing prealloyed Mo-Ti-C powders. Processing parameters were varied in an attempt to produce a composition similar to that of commercial Mo-0.5Ti alloy. This was successfully accomplished except for an abnormally high oxygen content. The oxygen present in the material caused compositional variations during subsequent processing and resided as a finely dispersed Mg-Ti-O phase.

The alloys, after sintering, were extremely fine grained and were readily hot and cold workable. A bend-transition temperature below -80°F was determined for one lot of material in the partially recrystallized condition. Tensile properties were similar to those of the commercial alloy at 2000°F ; however, the recrystallization temperature was considerably lower.

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CO-REDUCTION AND CONSOLIDATION OF Mo-0.5Ti-0.1C ALLOY

I. INTRODUCTION

The purpose of this research program has been to establish the feasibility of producing prealloy refractory metal powders. This was accomplished by co-reducing mixed halides of the alloying elements by a modified Kroll process.

Through the production of prealloyed metal powders it is felt that significant technical and economic advantages can be gained. Such processing could eliminate the necessity for primary consolidation by arc or electron-beam melting and would allow direct consolidation of complex alloys by powder metallurgy techniques. The economic advantage is obvious; however, benefits gained from grain size control, improved homogeneity, and the ability to directly produce finished parts are far more significant.

In this program, Mo-Ti-C alloys were produced by magnesium reduction of the preblended chlorides of these elements. This class of alloys was selected because of their commercial significance and the availability of physical and mechanical property data for the alloys made by conventional processing techniques.

Kroll processing was selected to accomplish our objectives because of its versatility and proven capability for producing most of the metals of interest as alloying additions to refractory metals.

II. EXPERIMENTAL PROCEDURE

A. Reactor Design, Handling, and Feed of Charge

The reactor design used for co-reduction of the mixed chloride feed was adopted from a previous design described by Block.⁽¹⁾ A schematic diagram of the reactor appears in Figure 1. It consists of two concentric shells; the upper one, which serves as a cover and contains the feed tube, fits into the lower such that its bottom surface is contained within

(1) Block, F.E., and Ferrante, M.J., "Vanadium by Metallic Reduction of Vanadium Trichloride," Journal of the Electrochemical Society, May, 1961.

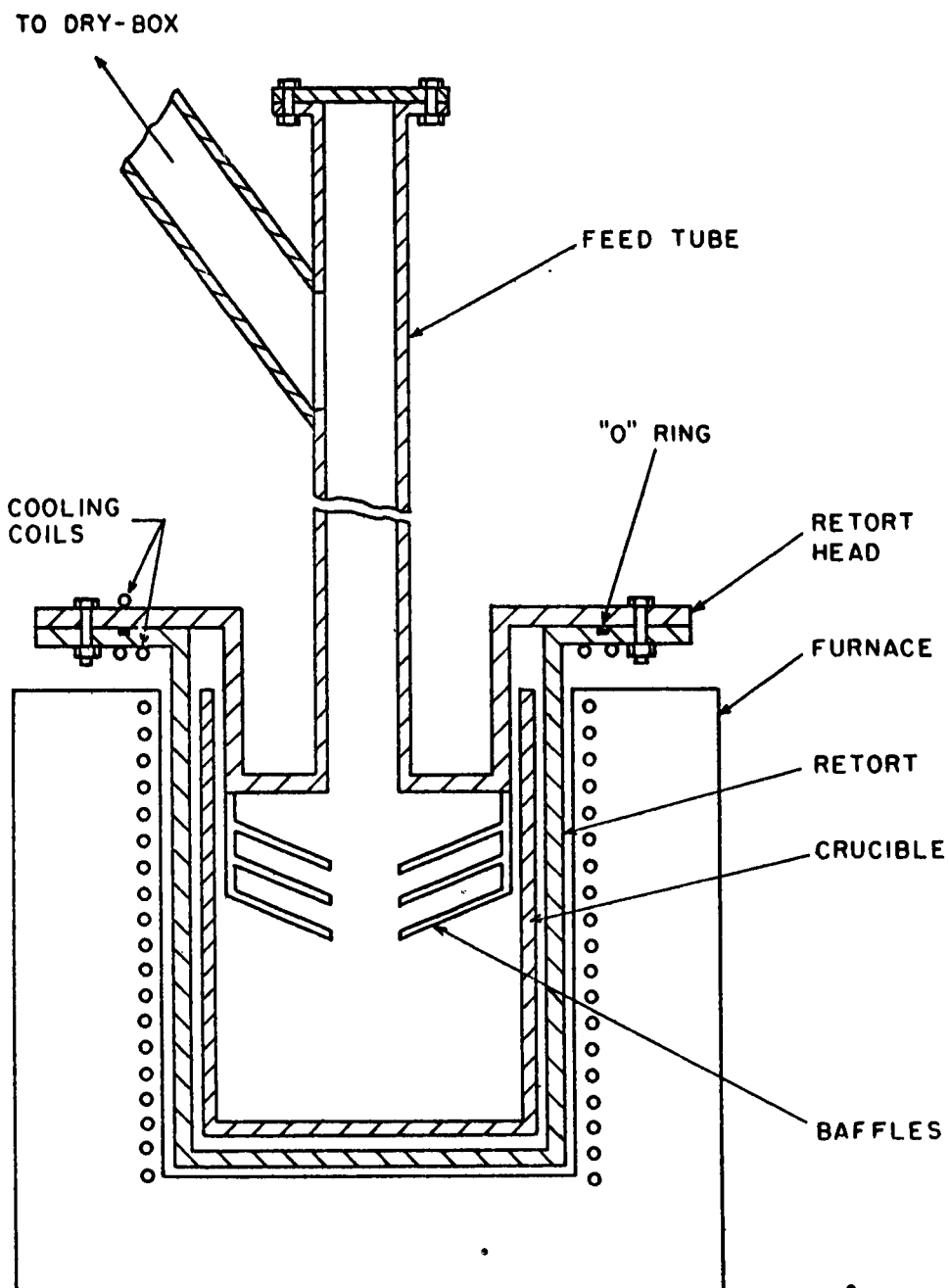


FIG.1 - EXPERIMENTAL KROLL REACTOR

the hot zone of the reaction furnace. This may be seen in Figure 2. Stainless steel baffles, having a 1-inch diameter opening, provide additional heat reflectors and serve to contain any volatilized reactants or reaction products. The reflector is sealed with a water-cooled O-ring gasket, protected from chloride attack by a stainless steel liner which fits snugly over the upper section of the reactor and extends to the bottom of the retort. A 5-inch diameter molybdenum crucible, containing molten magnesium, is located at the bottom of the retort.

Atmospheric control and feeding is accomplished by direct connection of the reactor to a dry-box as shown in Figure 3.

A typical run proceeds as follows: the metal chlorides in sealed containers and all of the equipment required for blending, packing, and feeding are placed in the dry-box. The dry-box is evacuated to a pressure of 10^{-3} mm Hg and then flushed with high-purity argon. This operation is repeated three times, and finally a static atmosphere of argon is established. The blended chlorides are then packed into magnesium tubes, 1/2-in. diameter x 1 1/2 in. long by means of a hand press. The ends of these tubes are slit to form five tabs which are bent over to form a reasonably tight seal. Each tube weighs about 2 grams and contains 7-8 grams of blended chlorides. These tubes are then hand fed through a pipe connected directly from the dry-box to the reactor. The quantity of reducing agent used in these reactions is derived from the amount of magnesium used for encapsulating plus an additional amount previously added to the reaction crucible.

B. Distillation

Upon the completion of a reduction run the reactor is sealed and allowed to cool. After cooling to room temperature the reactor is opened and the molybdenum crucible containing the reaction products is quickly transferred to the distillation unit shown in Figures 4 and 5. The distillation head is quickly sealed and the entire system evacuated with a high-capacity (15 cfm) roughing pump. The pressure is subsequently lowered to 5×10^{-6} mm Hg with the aid of 4-inch oil diffusion pump. Distillation is carried out at 1550° - 1600° F for 5-10 hours. These conditions are adequate for complete removal of the reaction by-products, Mg and $MgCl_2$, in the quantities that were present in our runs.

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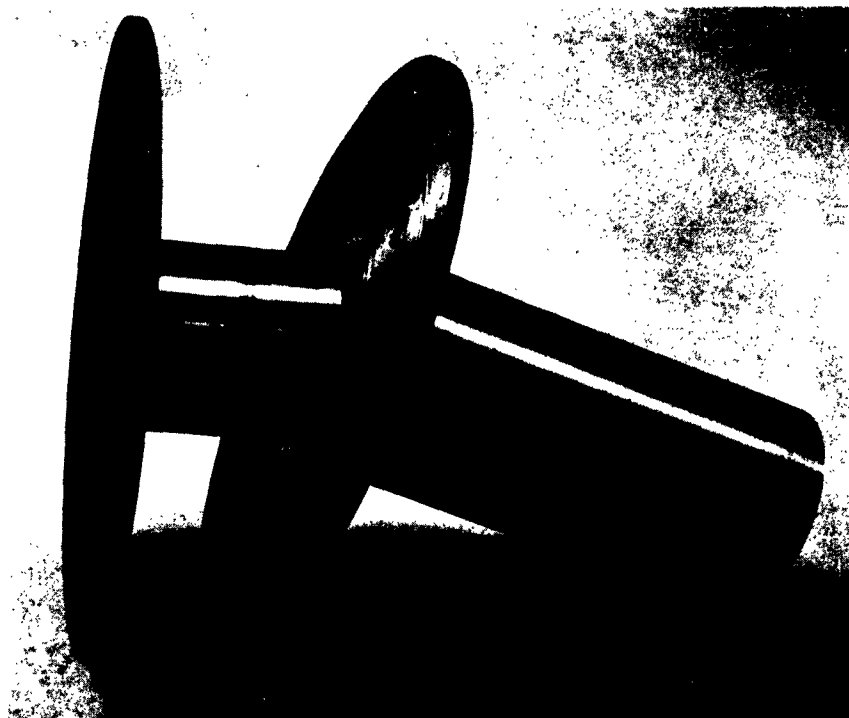


Fig. 2 - Basic Reactor Unit Consisting of Two Concentric Shells Which are Hermetically Sealed With a Water-Cooled O-Ring Gasket.

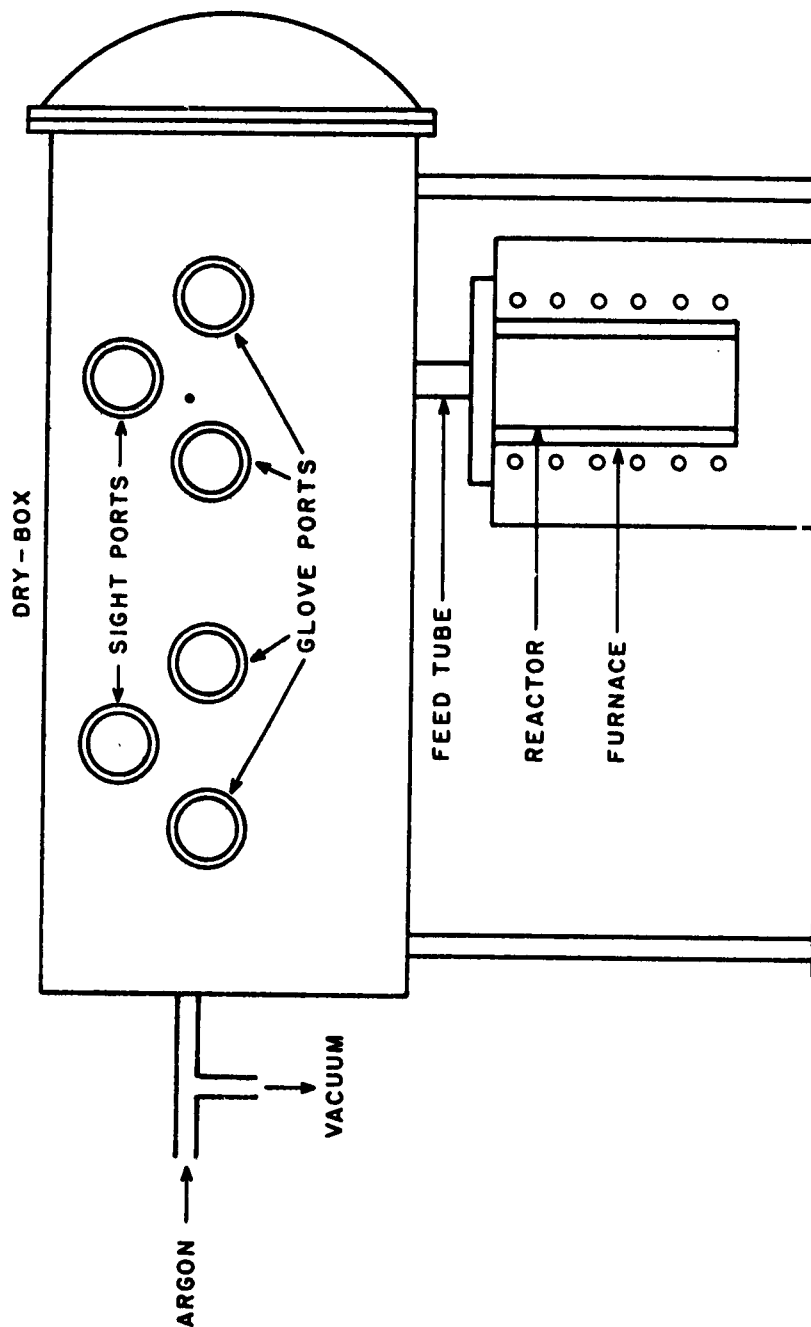


FIG.3 DRY-BOX AND REACTOR ARRANGEMENT.

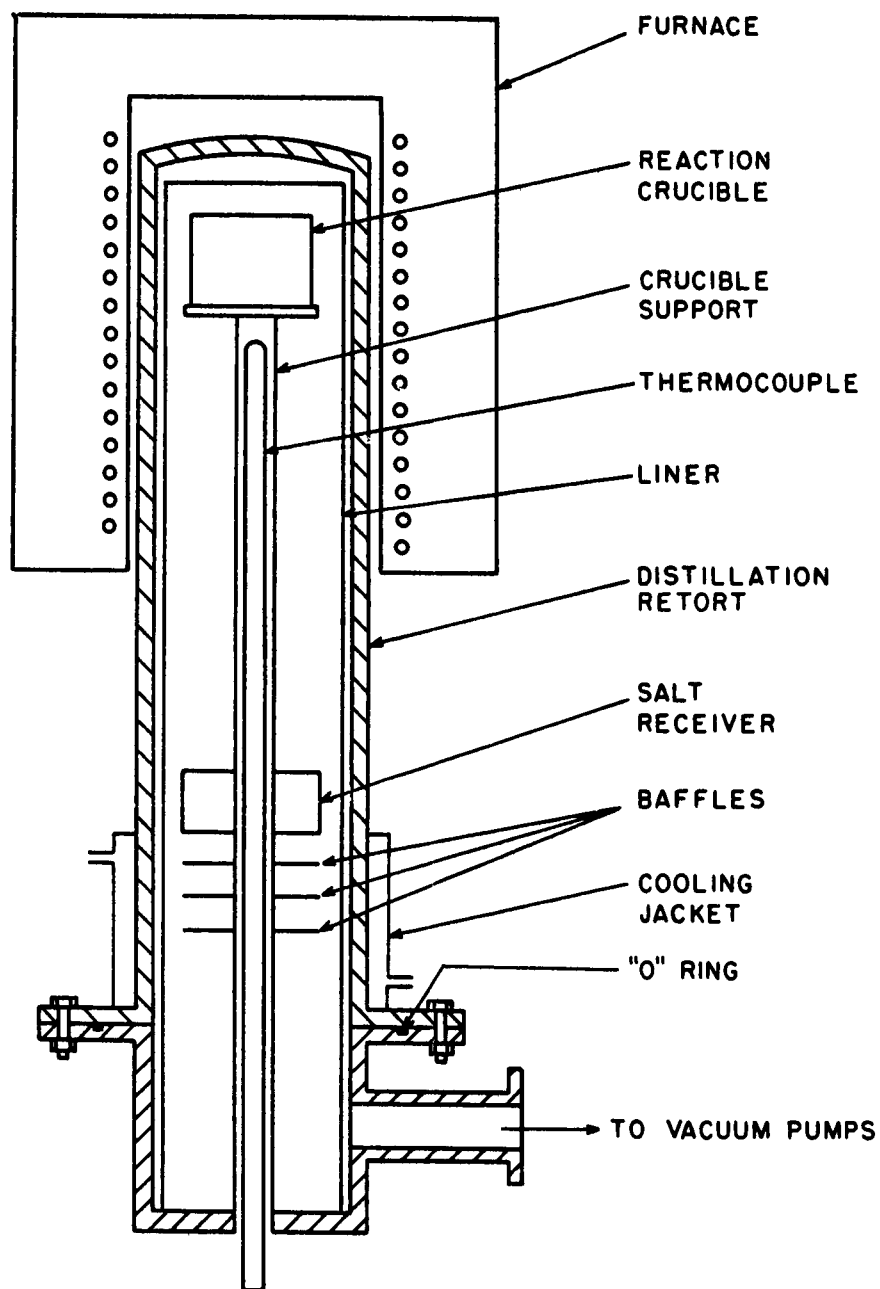


FIG. 4 - DISTILLATION - REDUCTION UNIT

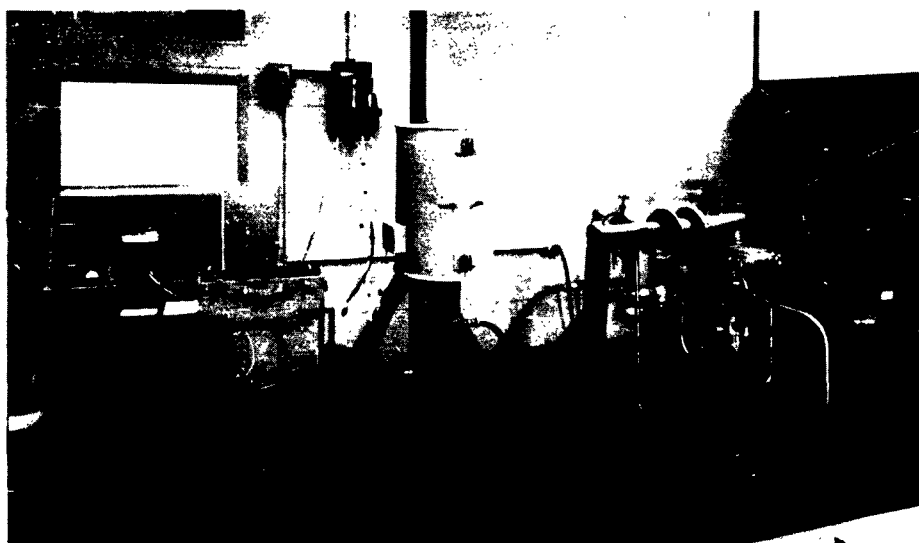
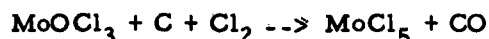


Fig. 5 - Distillation Unit Connected to Vacuum System.

C. Purification of MoCl₅

Commercially available MoCl₅ was purified by passing this material, in the vapor state, through activated charcoal in the presence of excess chlorine. The experimental arrangement is shown in Figures 6 and 7. Impure MoCl₅ is heated to 480°-570°F in the boiler shown on the left-hand side of the diagram in Figure 6. The vapor formed is blended with chlorine and passed through activated sugar-charcoal heated to 1290°-1650°F. Purification is accomplished primarily through the reaction:



The chlorine concentration is maintained at three times, or more, the stoichiometric requirement in order to help drive the reaction to the right and to avoid reversal of the reaction in the condenser section. The reaction products are passed through the condenser loops maintained at 375°-400°F where MoCl₅ is formed as a liquid. The liquid is drained into a bulb connected to the bottom of each loop and, when full, the bulbs are sealed with a torch by closing the Pyrex tubing connecting the bulb to the condenser. The upper part of the Pyrex tube is then sealed in order to contain a sample for chemical analysis. The bulb containing the purified pentachloride is transferred to the dry-box.

III. EXPERIMENTAL RESULTS

A. MoCl₅ Purification

The results of MoCl₅ purification experiments are presented in Table I. Several preliminary experiments were performed to establish the parameters which influence purification and to develop a satisfactory condensation and trapping system. This work was performed during runs number 1 through 6. After run No. 6 the equipment was modified to that shown in Figures 6 and 7.

The oxygen content of the commercially available MoCl₅ varied from 0.60 to 1.5 w/o. This was reduced to less than 0.01 w/o in runs 7 and 18, and to 0.008 w/o oxygen in run No. 8. The increasing oxygen

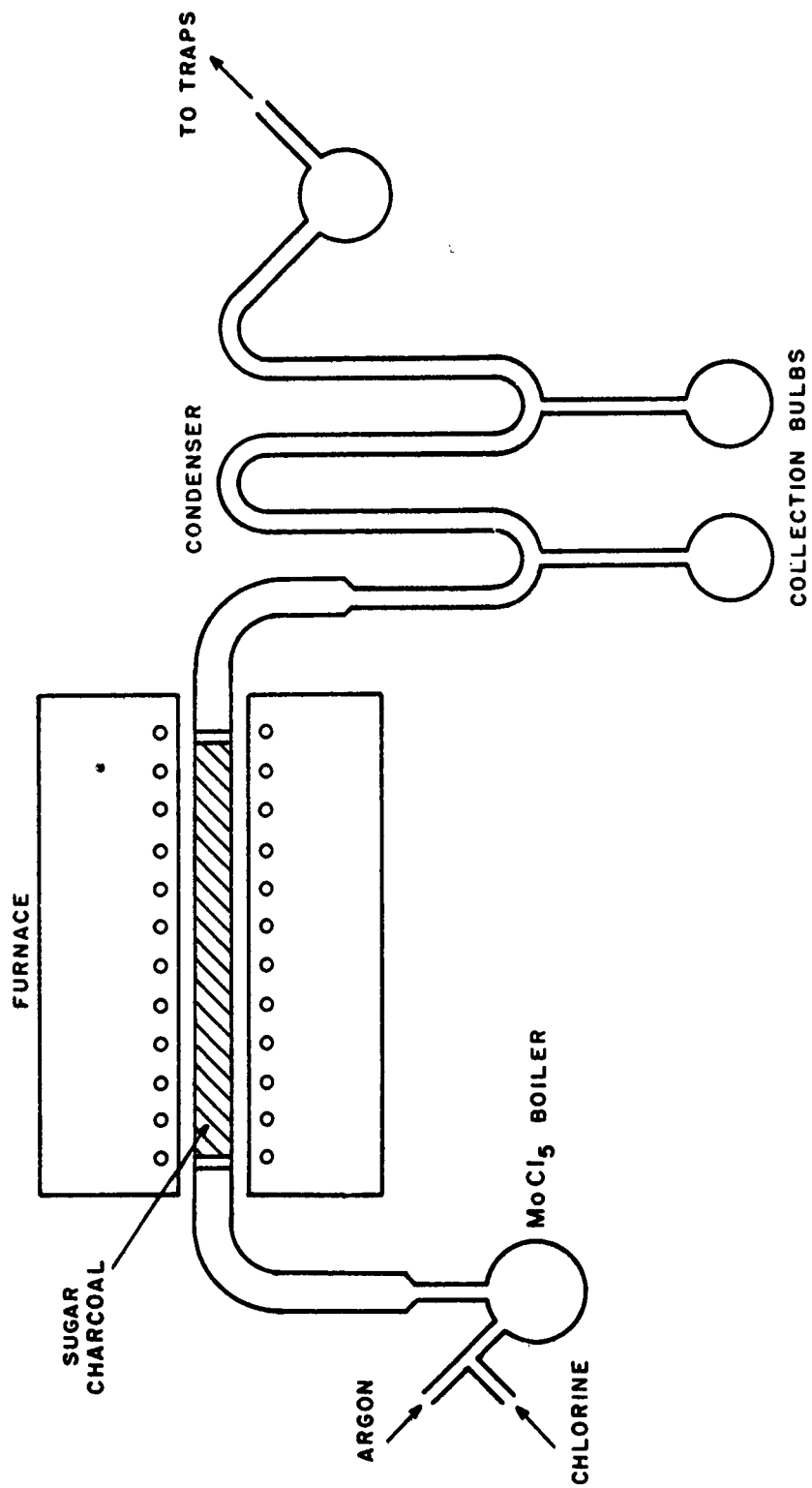
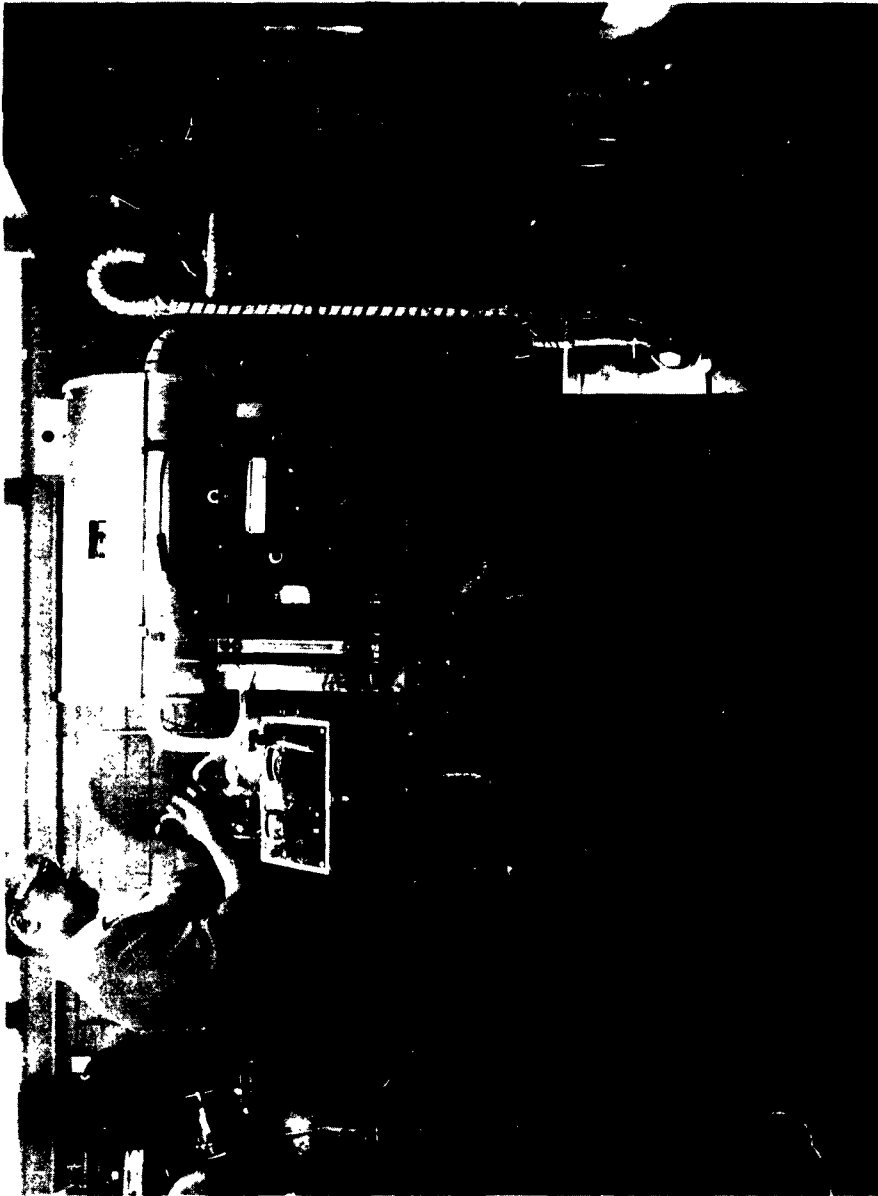


FIG.6 MoCl₅ PURIFICATION SYSTEM.



Neg. No. 22734

Fig. 7 - Photograph of MoCl₅ Purification Apparatus Shown Schematically in Fig. 6.

TABLE I
DATA FOR MoCl₅ PURIFICATION RUNS

Run No.	MoCl ₅ Distillation Rate, g/hr	Cl ₂ Flow Rate, cfh	Reduction Temp., °C	Condenser Temp., °C	Oxygen Content, w/o
1	No data taken - apparatus checked				
2	40	0.98	900	240	0.27
3	40	1.25	900	230	0.15
4	30	1.25	800	230	0.13
5	30	1.0	700	225	0.24
6	410	2.0	800	200	0.18
7	--	1.8	800	200	<0.01
8	470	2.5	800	200	0.008
9	380	2.0	800	200	0.02
10	--	1.5	800	200	0.03
11	455	3.0	800	200	0.05
12	490	2.5	800	200	0.06
13	490	2.5	800	200	0.10
14	485	2.5	800	200	0.16
15	460	2.5	800	200	0.09
16	568	2.5	800	200	0.27
17	550	2.5	800	200	0.47
18	540	2.5	800	200	<0.01
19	543	2.5	800	200	0.04
20	560	2.5	800	200	0.05
21	650	2.5	800	200	1.06
22	595	2.5	800	200	0.28
23	615	2.5	800	200	0.06

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content for runs 7 through 17 was apparently due to progressive deactivation of the charcoal used for purification. Deactivation could result from deposition of a stable material on the charcoal. After Run 17 the charcoal was analyzed and found to contain 0.09 w/o Mo and 0.01-0.05 w/o Cl.

In Run 18, the substitution of fresh charcoal produced by heating confectioners sugar to 800°F in a limited oxygen atmosphere, reduced the oxygen content of the purified pentachloride to less than 0.01 w/o. After each succeeding run the charcoal was reactivated by heating to 1100°F in chlorine. The high oxygen contents reported for runs 21 and 22 are apparently the result of poor sampling technique.

B. Reduction of Mixed Chlorides

1. Preliminary Experiments

Twenty-two reduction runs, of varying feed composition, were made. The total charge employed varied between 101.12 and 1,984 grams, the yield from the largest run being 612.2 grams of alloyed metal powder.

The first seven runs were performed with the objectives of determining the suitability of the equipment and of establishing the influence of reduction temperature and chemical species contained in the chloride charge on the properties of the reduced products. Subsequent runs had the primary objective of producing material of high purity.

Data for the preliminary runs, Nos. 2 through 7, appear in Table I. Run No. 1 was unsuccessful due to a jamming of the feed tube. Each successful run was made in argon at a pressure slightly greater than atmospheric. Pellets were fed to the reactor at a rate of two each minute. The product was, in each run, composed of loosely sintered powder aggregates. The amount of sintering and size of the aggregates was somewhat larger in the runs carried out at higher temperatures, although there was no significant variation in the average particle diameter of the product. A photograph of the reaction product contained in the reduction crucible after distillation appears in Figure 8. A photograph of one of the aggregates taken from the crucible is shown in Figure 9.

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Fig. 8 - Reaction Product of Run 1 after Distillation. Top View into Reduction Crucible.



**Fig. 9 - Aggregate of Sintered Reduced Alloy Powder Obtained
in Run 2.**

Processing data for the preliminary experimental runs appear in Table II. Each run will be discussed in greater detail in the following sections.

a. Run 2

A total chloride charge of 518.32 grams was prepared and reduced to metal at 1503° to 1597°F. The charge was designed to yield 185.3 grams of the alloy Mo-0.5Ti-0.1C upon reduction. The reduction was carried out in a 316L stainless steel crucible which resulted in serious iron and nickel contamination of the metal powder.

Twenty-gram samples of the product were consolidated by arc melting, by sintering in hydrogen at 3000°F for 2 hours, and by sintering in vacuum at 2200°F for 16 hours. Chemical analyses of the consolidated products are as follows:

Element	Per Cent in Product		
	Arc Melted	H ₂ Sintered	Vacuum Sintered
Oxygen	1.5-1.7	3.0-3.5	2.5-3.0
Carbon	0.035	0.033	0.002
Hydrogen	0.0008	0.019	0.0004
Titanium	<0.05	<0.05	<0.05
Iron	1.32	4.6	4.8
Magnesium (est.)	0.10	1.0	1.0

The high oxygen content is apparently the result of oxygen contained in the MoCl₅ feed. This material contains 0.60 w/o oxygen, probably present as MoOCl₃. The oxygen content of the product is about 3 w/o. The lower oxygen, iron, and magnesium content after arc melting is apparently the result of removal of these elements from the melt as a slag. During vacuum sintering, both oxygen and carbon were lost.

b. Run 3

Run 3 was made under conditions similar to those for Run 2 except that a titanium reduction crucible was used in place of stainless steel. The titanium content of the final product was 5.52 w/o.

TABLE II
SUMMARIZED DATA FOR KROLL REACTOR RUNS

Conditions	Run No.				
	2	3	5	6	7
Reaction temperature (°F)	1503-1597	847-914	897-914	866-884	847-866
Chloride charge (g):					
Total	518.32	1000 ^(a)	200	101, 12	101, 2
MoCl ₅	512.43 ^(b)	995 ^(b)	---	100 ^(c)	100 ^(c)
MoCl ₂	--	---	200	---	---
TiCl ₄	3.57	6.94	---	0.67	---
TiCl ₃	--	---	---	---	0.75
CCl ₄	2.32	4.52	---	0.45	0.45
Magnesium (g):					
As tubing	172	262	74	32	32
Original charge	200	400	200	122	119
% Excess reducing agent	215	177	840	594	580
Yield:					
Theoretical (g)	185.3	360	114.9	35.33	35.4
Actual (g)	186	325.5	107.5	25.6	22.1
%	100.4	<90.5	93.6	72.5	62.4
Reduction crucible material	316L SS	Ti	Ta	Mo	Mo

(a) Some charge was inadvertently lost during feeding.

(b) Commercially pure MoCl₅.

(c) MoCl₅ obtained from Alloyd Corp.

c. Run 5

In run 5, MoCl_2 was used as the feed material. Titanium and carbon additions were not made. Reduction was carried out in a tantalum crucible which became severely embrittled during the reaction. The oxygen content of the molybdenum produced was 1.2 w/o and magnesium, present as MgO was estimated at 0.1 w/o.

d. Run 6

In run 6, purified MoCl_5 , obtained from the Alloyd Corp. was used as the primary feed material. Analyses of the product after compacting and vacuum sintering at 1200°C for 16 hours show the following (in w/o):

Oxygen	1.0
Carbon	0.008
Titanium	0.07
Magnesium (est.)	1.0

The MoCl_5 was again checked as a source of oxygen contamination and found to contain 0.61 w/o oxygen. This oxygen is apparently accumulated during the time interval from which the purified MoCl_5 is first produced until it is reduced. The material when received at the Foundation was contained in a glass jar sealed with a taped cover. The jar was further sealed with wax; however, previous experience has shown that seals of this type are inadequate for protecting the highly reactive pentachloride from reaction with the atmosphere.

e. Run 7

Run 7 was made under conditions identical to those for Run 6 except that TiCl_3 was used in the feed in place of TiCl_4 . The product was pressed and sintered at 2200°F for 12 hours in vacuum. After sintering, the metal was found to contain 0.42 w/o Ti and 0.005 w/o C.

These preliminary experiments served to define tentatively the important processing variables necessary for the production of prealloyed powder. The necessity for performing the reaction in a molybdenum crucible and the desirability of replacing TiCl_4 with the less volatile TiCl_3

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were established. Temperature, over the range where this process could be feasibly carried out, did not significantly influence the character of the reduced powder.

The most serious experimental problem which remained to be overcome was elimination of the oxygen contamination derived from the starting materials and during handling of the feed. The presence of oxygen interferes with the co-reduction process and, when present in the quantities found in our alloys, exists as a stable oxide which is not removed during subsequent distillation. The oxygen also can account for the loss of carbon during sintering operations.

To reduce oxygen contamination, purified MoCl_5 (described in Section III-A) was used as the primary feed material and steps were taken to improve the quality of our dry-box atmosphere and general handling procedures.

2. Reduction of Purified MoCl_5

Runs 8 through 22 were performed with purified MoCl_5 as the primary feed material. The results of these runs are presented in Table III.

The oxygen content of the pentachloride feed varied for each run and was related to the oxygen content of the final product, although the oxygen contained in the final product was apparently derived from both the feed and as the result of contamination during processing. The lowest oxygen content, 0.19 w/o, occurred in Run 17 in which the MoCl_5 feed contained less than 0.01 w/o oxygen. The oxygen was present as a ternary oxide of magnesium and titanium after sintering at temperatures near 3600°F .

The titanium content of the runs in which TiCl_3 was used varied between 0.40 and 0.53 w/o, excluding Run ii. In Run 8 a sevenfold excess of TiCl_4 was used, which if quantitatively reduced would have yielded an alloy containing 3.5 w/o. The actual titanium content of this alloy was 1.5 w/o.

Carbon contents varied between 0.002 and 0.035 w/o, with most of the values falling between 0.002 and 0.004 w/o. The carbon contents

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TABLE III
REDUCTION RUNS PERFORMED WITH PURIFIED MoCl₅

Run No.	Quantity of Feed	Yield, grams	Composition, %			Purified MoCl ₅ Run No.
			Ti	C	O	
8	100 g MoCl ₅ 3.15 cc TiCl ₄ 0.30 cc CCl ₄	28.83	1.5	0.035	1.0+	6
9	367 g MoCl ₅ 2.75 g TiCl ₃ 1.10 cc CCl ₄	102.9	0.53	0.021	1.0+	7
10	553 g MoCl ₅ 0.3 g stearic acid 4.2 g TiCl ₃	174.8	0.45	0.004	0.64	8
11	571.1 g MoCl ₅ 0.3 g stearic acid 4.3 g TiCl ₃	193.4	0.12	0.003	0.94	9
12	777 g MoCl ₅ 0.5 g stearic acid 5.85 g TiCl ₃	254.1	--	--	--	11
13	1275 g MoCl ₅ 0.8 g stearic acid 9.6 g TiCl ₃	501.5	0.49	0.028	0.5	12 and 13
14	955.6 g MoCl ₅ 7.2 g TiCl ₃ 2.86 cc CCl ₄	280.2	0.44	0.002	0.70	15
15	1192.0 g MoCl ₅ 9.0 g TiCl ₃ 3.57 cc CCl ₄	295.6	0.40	0.003	0.90	17
16	1450.0 g MoCl ₅ 10.93 g TiCl ₃ 4.34 cc CCl ₄	Failure	--	--	--	16
17	1730 g MoCl ₅ 13.06 g TiCl ₃ 5.18 cc CCl ₄	561.1	0.47	0.003	0.19	18

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TABLE III (Cont.)

Run No.	Quantity of Feed	Yield, grams	Composition, %			Purified MoCl ₅ Run No.
			Ti	C	O	
18	1720 g MoCl ₅ 13.06 g TiCl ₃ 5.18 cc CCl ₄	515.7	0.44	0.004	0.46	19
19	1771.3 g MoCl ₅ 13.37 g TiCl ₃ 5.30 cc CCl ₄	544.1	--	--	--	20
20	1953.4 g MoCl ₅ 14.75 g TiCl ₃ 5.85 cc CCl ₄	612.2	--	--	--	21
21	1868 g MoCl ₅ 14.08 g TiCl ₃ 5.59 cc CCl ₄	569.1	--	--	--	22
22	1666 g MoCl ₅ 12.59 g TiCl ₃ 4.99 cc CCl ₄	504	--	--	--	23

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were determined from small cylindrical samples produced by compacting and sintering the reduced powder. Sintering was generally carried out at 2200°F in vacuum.

C. Consolidation and Fabrication of Prealloyed Powders

The material from reduction runs Nos. 10 through 15 was consolidated, individually, into 1-inch diameter bars by hydrostatic pressing. After pressing, the bars were sintered in hydrogen at 4000°F for 4 hours. The average diameter after sintering was 13/16 inch, and densities ranged between 89 and 93% of theoretical. The bars were sheathed in stainless steel jackets and rolled longitudinally at 2300°F. Small reductions were made and the bars were reheated after each pass. The objective of the "hot working" operation was to produce 0.5-inch thick flat bar stock for subsequent cold-working. This was successfully accomplished for the material of each reduction run except 10 and 11 which failed by cracking during the later stages of rolling.

After hot-working the sheathing was removed from the remaining bars and they were rolled, again longitudinally, in air at 1600°F. The bars were to be worked 90% at this temperature; however, this could only be accomplished for the materials of run 14 and 15. Run 13 was cold worked only 50%. The bar for Run 12 cracked after about 40% reduction.

The material for runs 17 through 22 was combined into two batches in order to produce larger ingots capable of being fabricated in a more conventional manner. Runs 17 through 19 were combined to form one batch weighing 1179 grams and the remaining powder--runs 20, 21, and 22--was combined to form a second batch weighing 1113 grams. Each batch was hydrostatically pressed into a 2-inch diameter bar and presintered in hydrogen at 3720°F for 135 minutes. The bars were subsequently sintered at 3900°F for 5 hours in vacuum. The densities after this treatment were 93.3% of theoretical for Batch 1 and 90.7% for Batch 2. An additional sintering at 4080°F for 3 hours in vacuum was used in an attempt to improve the density; however, no increase was obtained.

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The sintered bars were rolled longitudinally at 2300°F until a 25% decrease in thickness was accomplished. At this point they were cross-rolled, and after 44% decrease in the original thickness the bar for Batch 1 fractured into several pieces and that of Batch 2 cracked. A portion of Batch 2 was salvaged, and this material was rolled to strip at 1800°F. The total work at the lower temperature corresponds to 92% reduction in area.

D. Mechanical and Physical Properties

Alloys 13, 14, and 15, produced from the reduction runs listed in Table III and having the same numbers, were annealed for 1 hour at elevated temperatures to determine recrystallization behavior. The results appear in Figure 10. Alloy 13, which received only 50% cold work, has a recrystallization temperature between 2100° and 2200°F. The remaining alloys, cold-worked 90%, recrystallize, at least partially, below 2000°F. The alloys after recrystallization are very fine grained, the average grain diameter being about 10 microns. The microstructure of Alloy 13, recrystallized at 2200°F after 50% cold work, is shown in Figure 11.

Bend transition test specimens were made from the sheet produced for Run 15 and from Batch 2. Bending was performed over a 4t mandrel using the test and specimen preparation procedures outlined by the Materials Advisory Board.⁽²⁾ The specimens were annealed for 2 hours at 1900°F in vacuum prior to test. The hardness of the alloys after this annealing treatment was 255 VPN for Run 15 and 305 VPN for Batch 2. Run 15 was largely recrystallized. Alloy 15 has a bend transition temperature between -80° and -320°F. The specimens that were bent at room temperature and at the two lower temperatures are shown in Figure 12. The material produced from Batch 2 has a transition temperature above room temperature.

The tensile properties of the material from Run 15 and Batch 2 were determined at 2000°F in vacuum. The results are as follows:

(2) Report MAB-176-M, "Evaluation Test Methods for Refractory Metal Sheet Materials," Sept. 6, 1961.

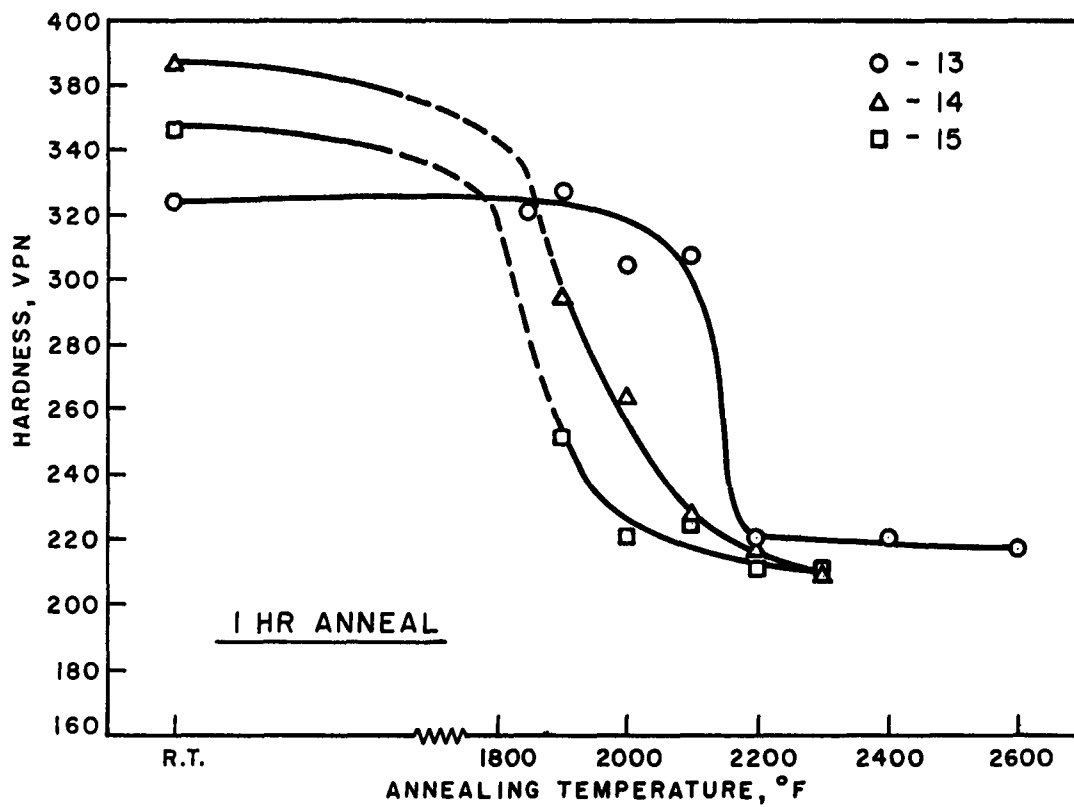
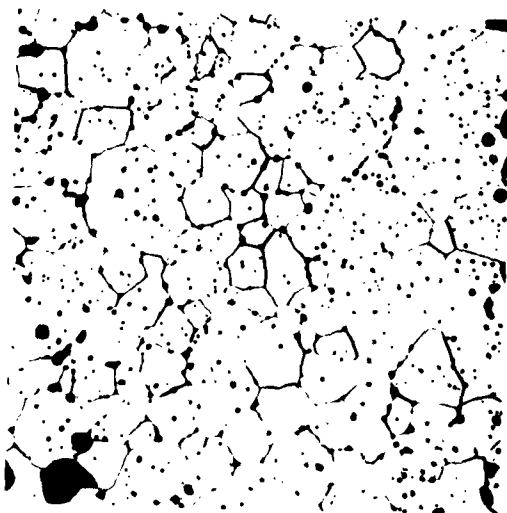


FIG. 10 RECRYSTALLIZATION CURVES FOR ALLOYS 13, 14, AND 15.



Neg. No. 23608

Mag. X1000

Fig. 11 - Microstructure of Run No. 13,
after Sintering at 3600°F and
Recrystallization at 2200°F
following 50% Cold Work.



Neg. No. 24001

Fig. 12 - Bend Transition Specimens of Alloy 15 Tested at -320° ,
 -80° , and $+75^{\circ}$ F.

<u>Alloy</u>	<u>0.2% Offset Yield Strength, psi</u>	<u>Ultimate Tensile Strength, psi</u>	<u>Elongation, % in 1 in.</u>
Run 15	37,200	47,000	20
Batch 2	48,500	54,500	9

The higher strength and lower ductility of Batch 2 is consistent with the greater oxygen content of this material. Microscopic examination reveals the presence of far greater insoluble, finely dispersed phase than that which exists in Run 15.

The hardness of the alloys after testing was 262 VPN for run 15 and 312 VPN for Batch 2.

IV. SUMMARY

A modified Kroll magnesium reduction process was used to produce prealloyed powder of Mo-Ti-C alloys. A total of 22 reduction runs was made. In most of these, molybdenum and titanium were nearly quantitatively reduced to metal. The use of TiCl_3 as a titanium source proved more satisfactory than the use of TiCl_4 for quantitative retention of the metal. Carbon, which was generally added to the feed as CCl_4 to produce 0.1 w/o C in the reduced metal, was retained in quantities of generally 0.03 w/o after reduction and distillation. Upon sintering in vacuum or hydrogen at elevated temperatures the carbon content was reduced to the vicinity of 0.003 w/o.

Early results indicated that oxygen contamination, derived from the feed materials and during processing, was the greatest experimental difficulty to be overcome. Consequently, molybdenum pentachloride purification procedures were developed and processing techniques were refined. Although pentachloride containing only 0.008 w/o oxygen was produced, the lowest oxygen content for the alloyed powder was 0.19 w/o.

The influence of oxygen on the reduction process was not investigated; however, its presence caused fine inclusions containing magnesium, titanium, and oxygen to be present in the final alloys. The presence of oxygen also caused the loss of carbon during vacuum or hydrogen sintering.

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The alloys were capable of being hot and cold worked, and have mechanical properties resembling those of other Mo-Ti-C or Mo-Ti-O alloys. The most comprehensive mechanical property data were obtained for the material produced in reduction Run 15. This alloy, after compacting and hydrogen sintering for 4 hours at 4000°F, contained 0.40 w/o Ti, 0.003 w/o C, 0.90 w/o O and a small quantity of magnesium combined with the titanium and oxygen. The recrystallization temperature for this alloy was close to 1900°F for material cold-worked 90%, and the bend transition temperature was between -80° and -320°F for material that was largely recrystallized. Tensile strength at 2000°F was 47,000 psi with an elongation of 20% for partially recrystallized material.

V. CONCLUSIONS AND RECOMMENDATIONS

The original objective of this program was to produce Mo-Ti-C prealloyed powder to be utilized for direct consolidation. Our objectives were hampered by contamination of feed materials with oxygen during handling and processing. As a result, the alloyed powder that was produced was contaminated with MgO which could not be removed during subsequent distillation and sintering operation. The presence of MgO in these materials apparently accounts for the loss of carbon during sintering and causes the titanium present to combine as an insoluble phase containing magnesium, titanium, and oxygen. Attempts to eliminate the presence of oxygen by purifying the feed materials and handling procedures were only partially successful. The lowest oxygen content which could be obtained was 0.19 w/o in reduction run No. 19.

Despite the presence of large quantities of oxygen, the alloys produced were generally readily fabricable, demonstrated a low transition temperature after partial recrystallization, and displayed good tensile strength at 2000°F. The recrystallization temperatures for these materials are lower than for commercial, arc-melted Mo-0.5Ti-C alloy; however, the material displays a very marked resistance to grain growth at high temperatures apparently due to the presence of the dispersed phase. Attempts to heat treat the alloys to cause solution and reprecipitation or work directed toward controlling the size and distribution of the dispersed phases was not performed

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but would provide an interesting study. However, it is recommended that future work in this area could be more satisfactorily extended by developing prealloyed powders of more refractory materials by Kroll processing. Alloys of tungsten and tantalum, the halides of which are less sensitive to oxygen contamination than MoCl_5 , should be produced by this technique. The grain size and homogeneity control which could be realized might lead to the development of a class of superior refractory metal alloys.

VI. CONTRIBUTING PERSONNEL, LOGBOOKS,
AND ACKNOWLEDGMENTS

The following personnel have contributed to this experimental program:

C. R. Simcoe, Supervisor
J. J. Rausch, Senior Metallurgist
J. E. Anderson, Technician
R. N. Chaney, Technician
D. E. Lofton, Technician

Data for this work appear in ARF Logbook Nos. C 11521, C 12048, C 12225, C 12431, and C 12923. Chemical analyses were performed on the reduced metal by Dr. K. A. Waehner of the Fansteel Metallurgical Corporation, and on the molybdenum chlorides by Dr. J. F. Below of the Stauffer Chemical Company.

Respectfully submitted,

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Tech Rev. - FCH
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